

# The exploitation of thin film coatings for fibre sensors for the application of chemical sensing

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## ABSTRACT

We report on the use of thin film coatings, both single and multi-layered, deposited on the flat side of a lapped, D-shaped fibre to enhance the sensitivity of two kinds of surface plasmon resonance based optical fibre sensors. The first kind involves the use of a tilted Bragg grating inscribed within the fibre core, prior to fibre coating, while the second relies on a surface relief grating photoinscribed after the fibre has been coated. Some of the devices operate in air with high coupling efficiency in excess of 40dB and an estimated index sensitivity of  $\Delta\lambda/\Delta n = 90\text{nm}$  from 1 to 1.15 index range showing potential for gas sensing. Other sensors produced index sensitivities ( $\Delta\lambda/\Delta n$ ) ranging from 6790nm to 12500nm in the aqueous index regime. The materials used for these fibre optical devices are germanium, silica, silver, gold and palladium.

Keywords: Index Sensing, Coatings, gratings, surface plasmon resonances

## 1. INTRODUCTION

There has been a strong interest in recent years in using gratings in fibres to produce chemical sensors, including long period gratings (LPGs) [1, 2, 3], fibre Bragg gratings (FBGs) and tilted fibre Bragg gratings (TFBGs). At present the majority of these sensors are used to detect the heavier organic/inorganic compounds, such as detection of organic aromatic compounds in paraffin [3]. Whilst the index of sensitivity of most grating based devices is highest for solutions that have an index of approximately 1.44, which is good for various organic hydrocarbon compounds, there is a need to increase sensitivity of these devices to aqueous solutions for biochemical applications, where index values typically range from 1.333 to 1.380 (aqueous index regime); for example, the effective index of a cell is about 1.36 to 1.38 [4,5]. Such sensors would also be appropriate for the detection of the lighter hydrocarbon compounds used for Aviation fuel [6] and if their response could be enhanced at even lower refractive indices, for gas detection. Another type of sensor based upon surface plasmon resonance (SPR) is showing the potential to yield high index sensitivities. Surface plasmon resonance is an important optical phenomenon that involves a resonant transfer of the incident light energy to a surface-plasmon (SP) mode in the form of collective electron oscillations in a metal [7]. The plasmons exist at a metal-dielectric interface and obey the following dispersion relation for two homogeneous semi-infinite media [8]:

$$\beta = k \sqrt{\left( \frac{\epsilon_m \cdot n_s^2}{\epsilon_m + n_s^2} \right)} \quad (1)$$

where  $k$  is the free space wave number,  $\epsilon_m$  is the dielectric constant of the metal ( $\epsilon_m = \epsilon_{mr} + i \epsilon_{mi}$ ) and  $n_s$  is the refractive index of the dielectric.

The need for high sensitivity at low indices is being addressed with the use of tilted Bragg gratings to assist in the generation of surface plasmon resonances in a fibre configuration [9, 10]. There is very little published with regards to multi-layered thin film SPR fibre devices, and the majority of these papers, such as [11] and [12], address the SP's optical properties and not their sensing potential.

In this paper, we report on two novel SPR fibre devices whose spectral sensitivities can be modified by using various coatings, in either single or multiple layers. A general observation of these devices is that they have polarisation dependence; the spectral location of maximum coupling to the SPR is highly dependent upon the polarisation state of the illuminating light and can be tuned from 1100nm to 1700nm.

Firstly, we describe a fibre device utilising a TFBG to enhance the coupling of the illuminating light to a SP generated on a coated, lapped single mode fibre [9,10]. It was observed that the spectral location of maximum coupling to the SPR was highly dependent upon the polarisation state of the illuminating light and could be tuned from 1100nm to 1700nm, thus producing various SP probe depths, dependent upon the polarisation. It was found that for a device that is still to be fully optimised, the maximum spectral index sensitivity ( $d\lambda/dn$ ) was 3365 nm for the index range 1.335 to 1.370 with coupling strength in excess of 25dBs.

Secondly, we describe a fibre device based upon a surface relief grating-type structure inscribed with UV light into a multi-layered thin film deposited on the flat side of a lapped, D-shaped fibre. The single layered devices were fabricated from germanium, while the multilayered ones comprised layers of germanium, silica, silver and gold. Some of the devices operated in air with high coupling efficiency in excess of 40dB and an estimated index sensitivity of  $\Delta\lambda/\Delta n = 90\text{nm}$  from 1 to 1.15 index range while others provided an index sensitivity of  $\Delta\lambda/\Delta n = 12500\text{nm}$  for refractive indices from 1.33 to 1.39.

## 2. FABRICATION AND CHARACTERISATION

The fabrication of the first type of SPR fibre device begins with the inscription of a grating in a UV photosensitive single mode fibre (hydrogenated standard telecommunications fibre) using a uniform phase mask (mask period = 1.0157 $\mu\text{m}$ ) mounted on a goniometer, tilted to a specific angle. Labels are added to indicate the orientation of the tilted grating. Next, the fibre is lapped down to 10 $\mu\text{m}$  from the core-cladding interface; it has been estimated that the error associated with this lapping is approximately  $\pm 1 \mu\text{m}$ . The labels on the fibre are used to indicate which region of cladding is to be removed so that the grating vector, the fibre axis and the normal to the lapped surface all lie in a plane. Thirdly, the flat of the lapped fibre is then coated with either gold or silver layer with a thickness of 38nm and 35nm respectively, using a sputter machine and mask.

The second type of SPR device is fabricated by firstly mechanically lapping a standard SMF fibre down to 10 $\mu\text{m}$  from the core-cladding interface. Secondly, using RF sputtering, a series of coatings are deposited upon the flat of

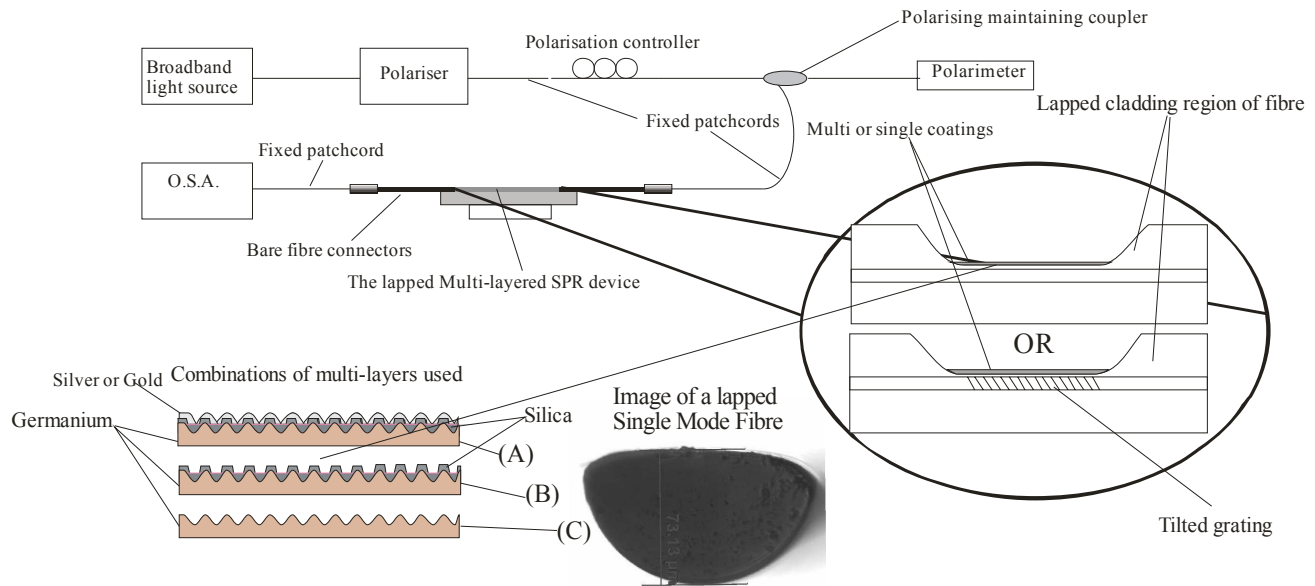
the lapped fibre. These coatings consisted of various numbers of layers and materials used such as germanium, silicon dioxide, silver and gold as described below. Thirdly, the coated fibre was exposed to a UV light interference pattern produced by a uniform phase mask with period  $1.018\mu\text{m}$  through laser beam scanning and multi-exposure. This produced a surface relief structure which has dominant spatial periods of  $\sim 0.5\mu\text{m}$  and  $\sim 1\mu\text{m}$ . A series of fibre devices were investigated that consisted of various coatings with the two coupling mechanism that produce the surface plasmon resonances, see table 1.

**Table 1** Materials and their thickness used in the fabrication of SPR fibre devices

| Sensor Type                               | Materials used<br>in the<br>construction | 1 <sup>st</sup> layer | 2 <sup>nd</sup> layer | 3 <sup>rd</sup> layer |            |
|---|--|-----------------------|-----------------------|-----------------------|------------|
|   |  | Germanium<br>nm       | Silica<br>nm          | Silver<br>nm          | Gold<br>nm |
| UV grating inscribed<br>in the fibre core | Device Ag Tilted                         | -                     | -                     | 35                    | -          |
|   | Device Au Tilted                         | -                     | -                     | -                     | 38         |
|   | Device Au Tilted                         | 48                    | 48                    | -                     | 32         |
| UV post processed<br>coating              | Device Ge overlay                        | 48                    | -                     | -                     | -          |
|   | Device SiO <sub>2</sub> overlay          | 48                    | 48                    | -                     | -          |
|   | Device Ag overlay                        | 48                    | 48                    | 32                    | -          |
|   | Device Au overlay                        | 48                    | 48                    | -                     | 32         |

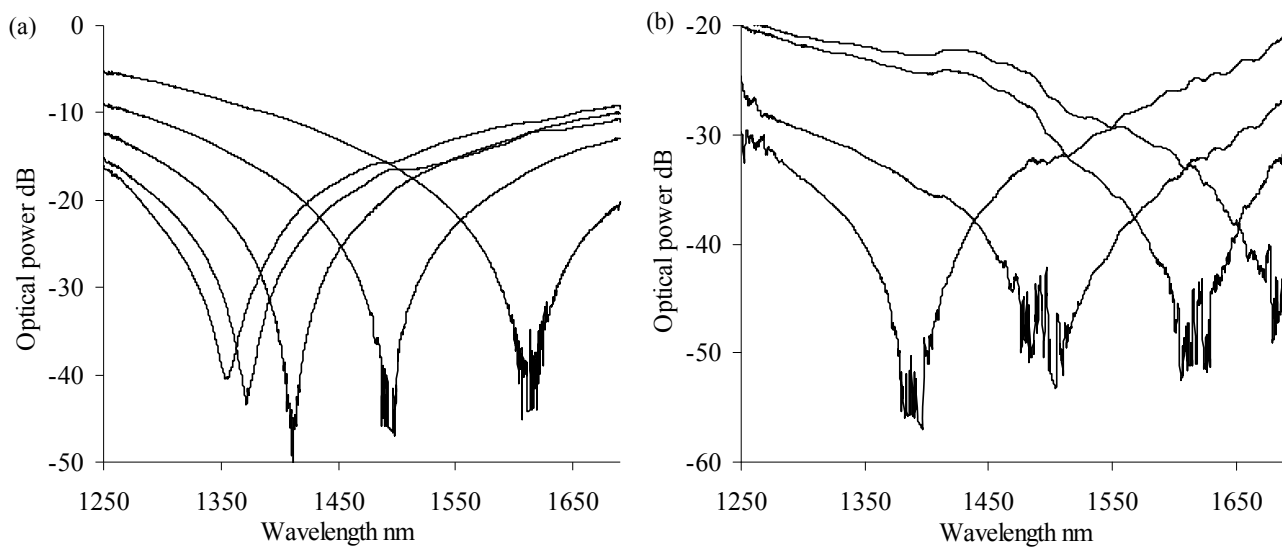
The rationale for using these materials is in two parts. The first concerns the optical constants of the materials and the requirement that their dispersion relationships must allow coupling to surface plasmons at a metal–dielectric or semiconductor–dielectric interface; both Ge and Ag exhibit this behaviour. Secondly, Ge and SiO<sub>2</sub> layers are used due to the fact that it is known from studies of grating formation [12] that when exposed to UV light, Ge/GeO produces photo-bleaching and compaction of the material, thus producing a surface corrugation on the multi-layered structure.

Prior to UV processing the polarisation dependence had been investigated and a small variation in the overall transmitted optical power with polarisation was found. After UV exposure the devices were further characterised by observing the spectrum of the transmitted light as the azimuth of the polarisation state was changed. These devices now showed a significant variation with polarisation, which is discussed in reference 10. To do this characterisation, light from a broadband light source was passed through a polariser and a polarisation controller before illumination of the sample, with the transmission spectra being monitored using an optical spectrum analyser (accuracy of  $0.005\text{nm}$ ). The change in polarisation of the illuminating light was monitored with a polarimeter (Tektronix, PAT 9000B) via a polarisation maintaining coupler, see figure 1.



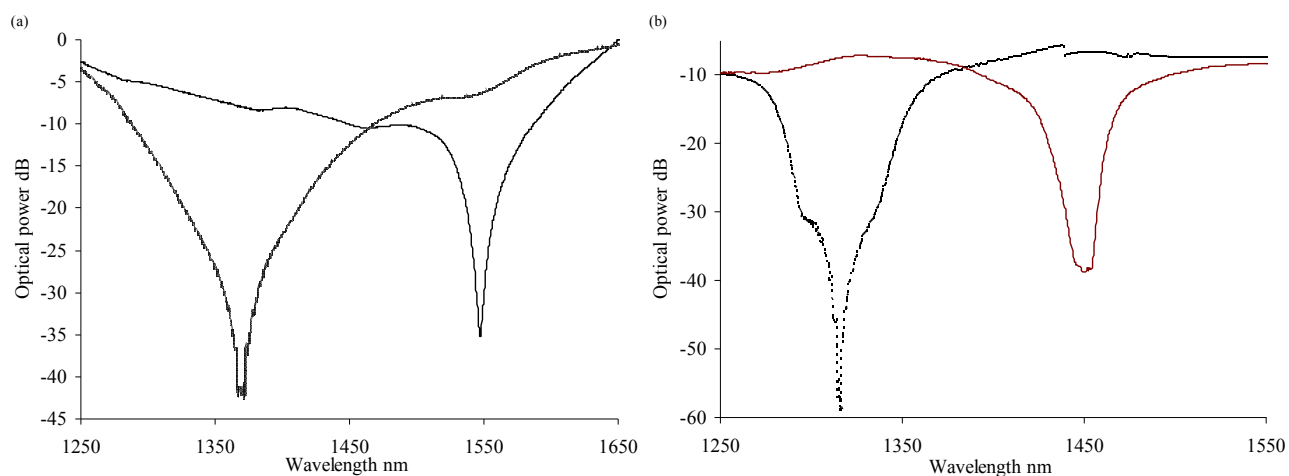
**Figure 1.** Scheme used for the characterisation of the lapped and multi or single-layer coated fibre devices; typical examples of combinations of layers used in the samples (A) Ge-SiO<sub>2</sub>-(Ag or Au), (B) Ge-SiO<sub>2</sub>, (C) Ge; a typical cross-section of the devices.

All the fibre devices showed polarisation dependence but the spectral responses differed between the two types of devices. Firstly, the tilted Bragg grating devices could be tuned and produced large extinction ratios over a very wide wavelength range; this occurred for either single or multi-layered coatings, see figure 2.



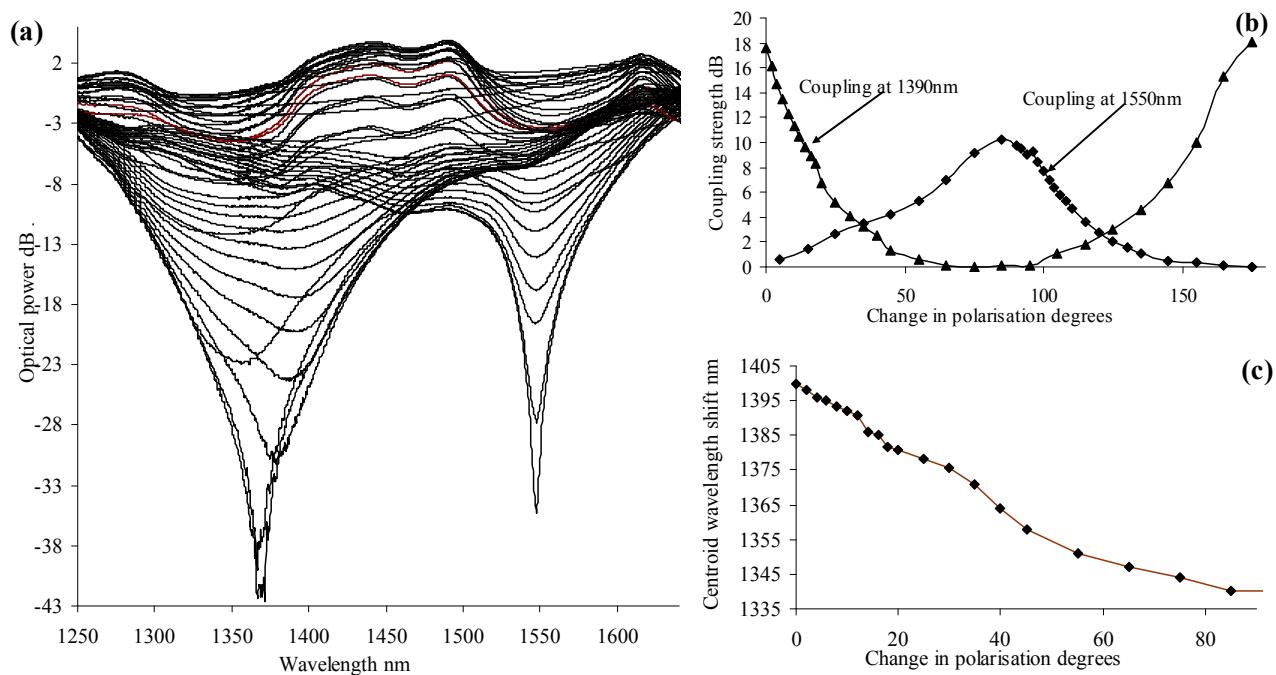
**Figure 2** Evidence of surface plasmon resonances obtained at different wavelengths by varying the polarisation of the illuminating light on one device, (a) Au-TFBG, (b) Au-SiO<sub>2</sub>-Ge-TFBG. Both devices had a 7 degree tilt angle and were submerged in a medium with a refractive index of 1.36.

Varying the polarisation for the surface relief type SPR fibre devices produced two resonances over the observed wavelength range, see figure 3.

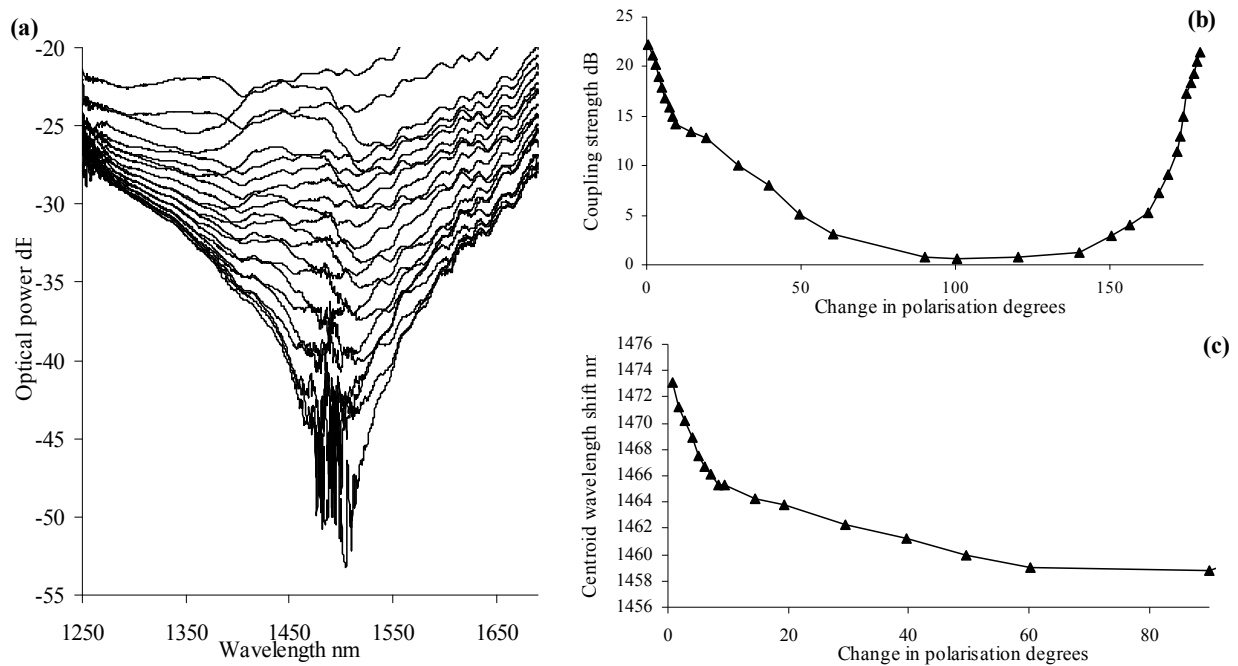


**Figure 3** Transmission spectra of the Ge-SiO<sub>2</sub>-Ag (a) and Ge-SiO<sub>2</sub> (b) coated fibre devices surrounded by air for two polarisation states of the illuminating light

It was also observed that polarisation sensitivity varied from device to device investigated, figure 4 and figure 5 show typical examples of the sensitivities of these devices



**Figure 4** (a) Transmission spectra of the Ge-SiO<sub>2</sub>-Ag coated surface relief fibre device as a function of changing polarisation from maximum coupling in air. (b) The coupling efficiency of the resonances at 1390nm and 1550nm and (c) the wavelength shift of the coupling feature at 1390nm as the azimuth of polarisation of the illuminating light is changed from the position providing maximum coupling.

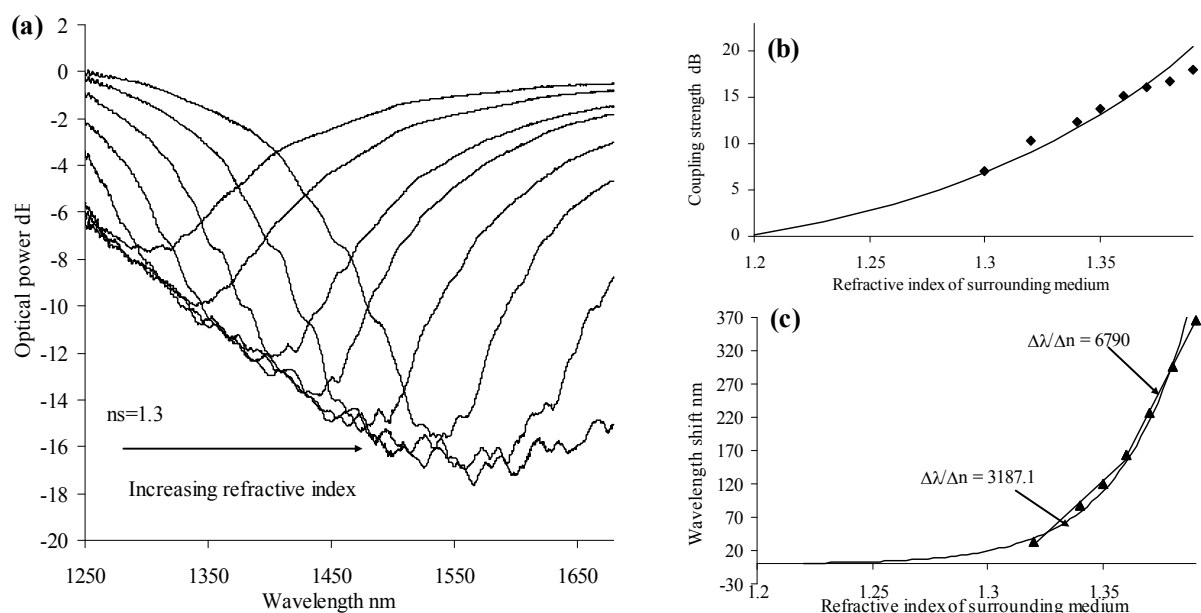


**Figure 5** (a) Transmission spectra of the Ge-SiO<sub>2</sub>-Au coated tilted Bragg fibre device as a function of changing polarisation from maximum coupling with a surrounding medium of 1.36. (b) The coupling efficiency of the resonance at 1480nm and (c) the wavelength shift of the coupling feature at 1480nm as the azimuth of polarisation of the illuminating light is changed from the position providing maximum coupling

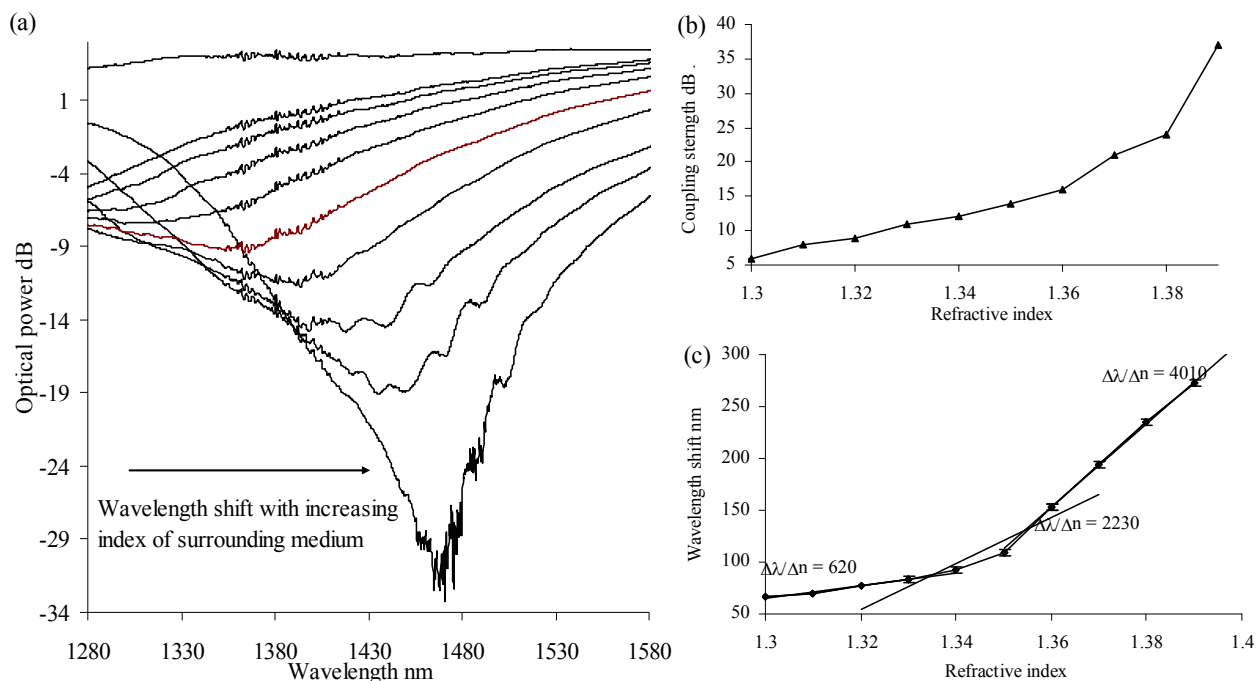
We are mostly interested in the potential of these SPR devices for chemical and biochemical sensing applications using wavelength and intensity interrogation. Whilst it may seem that the polarisation sensitivity of these SPR devices may be problematic, this can potentially be overcome by using polarisation maintaining fibre [13], or can be seen as another spectral property to be exploited. A detection scheme that does exploit this behaviour is angular interrogation [14]. Inspecting the transmission spectra in figures 2 and 5, there is no significant observed spectral feature associated with Bragg reflection from the gratings themselves. This is expected due to the fact their transmission profiles are very weak and that the interrogating light source is broadband and swamps the response.

### 3. REFRACTIVE INDEX SENSITIVITY

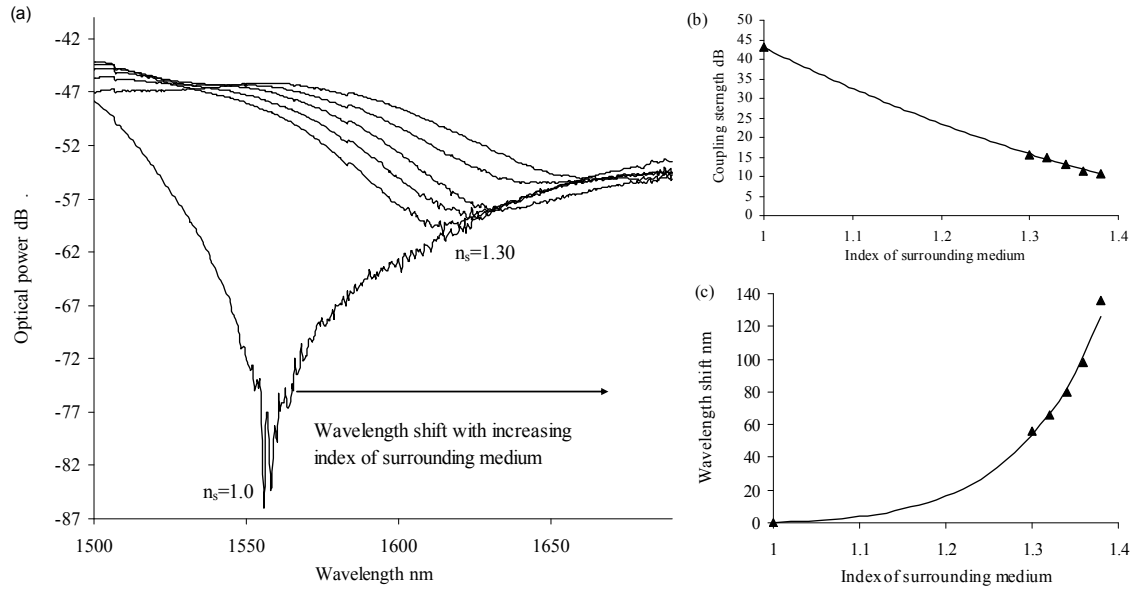
For refractive index sensitivity measurements the devices were placed in a V-groove and immersed in certified refractive index (CRI) liquids (supplied by Cargille laboratories Inc.) which have a quoted accuracy of  $\pm 0.0002$ . The devices and V-groove were carefully cleaned, washed in ethanol, then in deionised water and finally dried before immersion into the next CRI liquid. The V-groove was made in an aluminium plate, machined flat to minimise bending of the fibre. The plate was placed on an optical table, which acted as a heat sink to maintain a constant temperature. The V-groove was used in conjunction with the apparatus shown in figure 1. Figures 6 to 9 show some typical spectral index sensitivities measured for the devices.



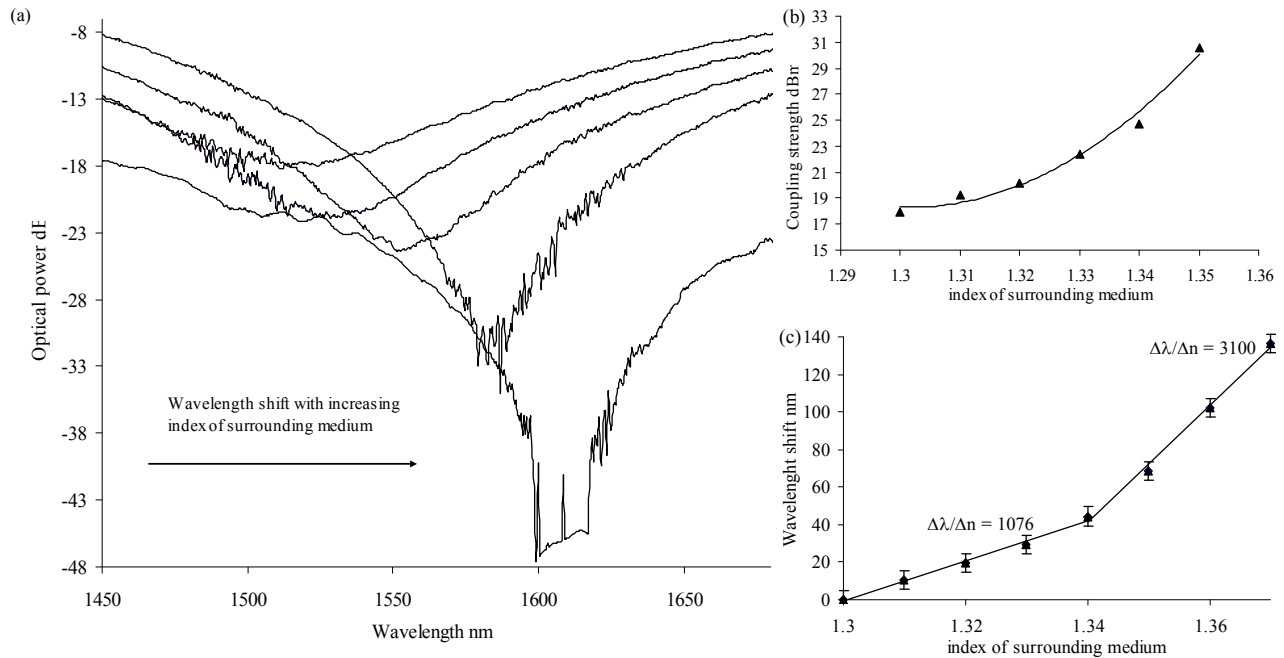
**Figure 6.** (a) Transmission spectra of the Ge coated surface relief device as a function of refractive index (polarisation of the illuminating light chosen to maximise coupling at index 1.3). (b) Coupling strength and (c) Wavelength shift of the resonance as a function of refractive index.



**Figure 7** (a) Transmission spectra of the Ge-SiO<sub>2</sub>-Au coated surface relief device as a function of refractive index (polarisation of the illuminating light chosen to maximise coupling at index 1.36). (b) Coupling strength and (c) Wavelength shift of the resonance as a function of refractive index.



**Figure 8** (a) Transmission spectra of the Ge-SiO<sub>2</sub>-Ag coated surface relief device as a function of refractive index (polarisation of the illuminating light chosen to maximise coupling at index 1.36). (b) Coupling strength and (c) Wavelength shift of the resonance as a function of refractive index.



**Figure 9** (a) Transmission spectra of the Ag coated tilted Bragg device (7 degrees) as a function of refractive index (polarisation of the illuminating light chosen to maximise coupling at index 1.36). (b) Coupling strength and (c) Wavelength shift of the resonance as a function of refractive index.

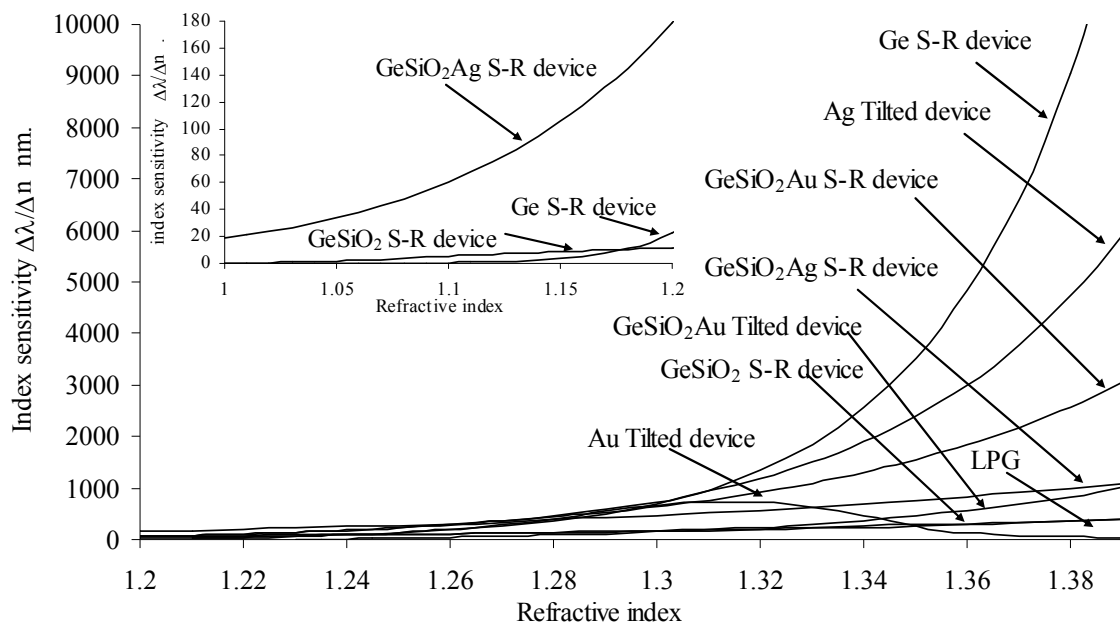
The highest wavelength spectral sensitivity to index was obtained with the Ge coating, which provided  $\Delta\lambda/\Delta n$  ranging from 3200 nm to 12500nm, see figure 6. Some the devices exhibited strong coupling in air such as the Ge-SiO<sub>2</sub>-Ag surface relief device which shows a decrease in coupling strength with increase in index, varying from 40dB in air to 7dB with a surrounding index of 1.39 and having a spectral sensitivity ranging from 710nm to 1200nm in the aqueous regime. The Ge-SiO<sub>2</sub>-Au surface relief device gave index sensitivities ranging from 2000nm to 4500nm with increasing



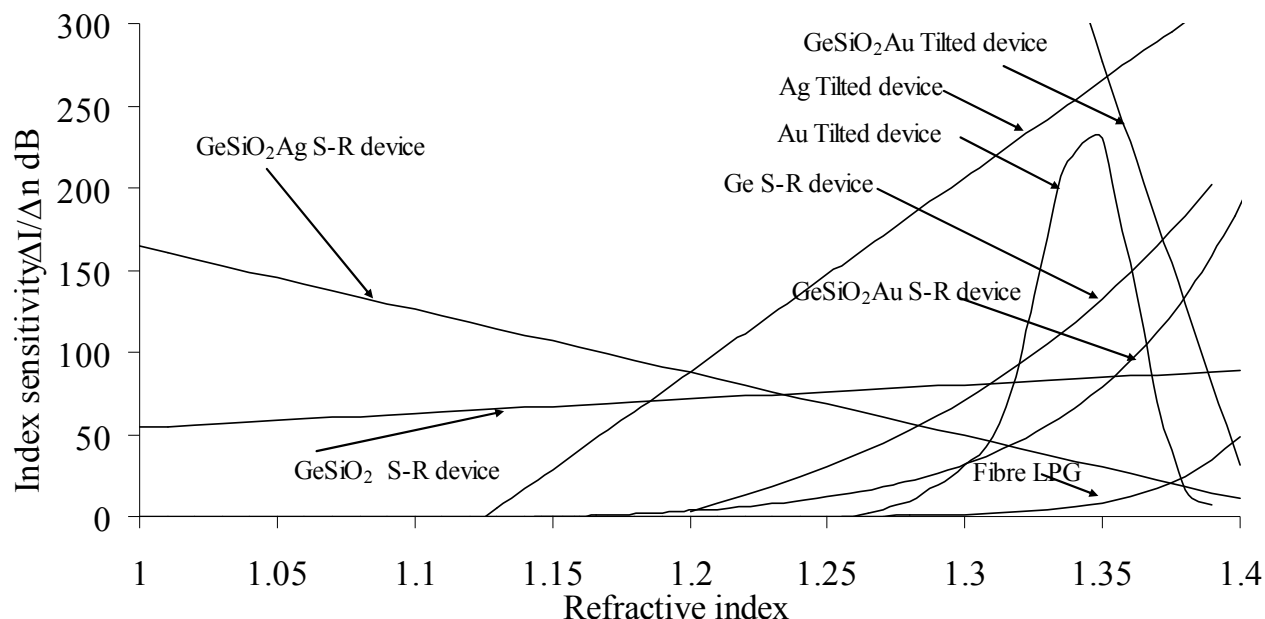
coupling strength from no coupling to 37dB. Considering the low index regime ranging from 1 to 1.15, the gold overlay fibre showed no coupling but the Ge-SiO<sub>2</sub>-Ag and Ge-SiO<sub>2</sub> gave promising results with wavelength shifts of 90nm and 40nm along with 30dB and 17dB changes in optical coupling from index 1 (air) to 1.3, respectively. The differently configured SPR devices yielded different responses for various index regimes results with some out-performing others for a given index range. This behaviour is expected due to the different dispersion relationships for each of the materials used in the coatings along with how the surface plasmons are supported by the different physical structures of the coatings.

To obtain an estimate of the spectral sensitivity of these SPR devices for low refractive indices from 1 to 1.1 we use the approach given in REF 15, which is to calculate the propagating modes of a D-shaped fibre with coating using a conformal mapping technique and then implement Fresnel's equations for a four layered system. The optical constants used in Fresnel's equations were estimated by an effective medium approximation, the Maxwell-Garnett theory [16], which yields an effective dielectric function as a function of the fractional volume of the metal/semi-conductor within an effective layer. In this work the effective change of the surrounding index is determined within a sensing volume, which is the area of the coating multiplied by the spatial extension of the evanescent field associated with the surface plasmon perpendicular to the coating's surface; a more detailed description is given in REF 6 and REF 11.

The experimental and theoretical results for wavelength and optical power spectral sensitivities are combined and shown in figures 10 and 11. These results are compared to a long period grating with a period of 240µm and a length of 5cm inscribed into standard single mode telecoms fibre. To attach any worth to these fibre sensors, their performance has to be compared to other sensor types in terms of wavelength shift (spectral sensitivity) and coupling strength variation (intensity sensitivity) as a function of index change. These results suggest the devices have potential in the low index regime and should demonstrate dramatic improvement in sensitivity/performance over LPG fibre devices in the aqueous index regime.



**Figure 10** Simulated and experimental wavelength spectral sensitivity comparison of some of the coated fibre devices (Tilted devices; SPR fibre devices that have a tilted Bragg grating written into the core of the fibre, S-R devices; SPR fibre devices with a surface-relief corrugation of the fibre coatings) as a function of refractive index along with a long period grating (period = 240µm, length 5cm).



**Figure 11** Simulated and experimental optical power sensitivity comparison of some of the coated fibre devices (Tilted devices; SPR fibre devices that have a tilted Bragg grating written into the core of the fibre, S-R devices; SPR fibre devices with a surface-relief corrugation of the fibre coatings) as a function of refractive index along with a long period grating (period = 240 $\mu$ m, length 5cm).

#### 4. CONCLUSION

We have investigated two types of surface plasmon resonance fibre devices with coupling mechanisms based upon either tilted fibre Bragg gratings or a surface relief structuring of the coating itself produced by ultra-violet exposure through a conventional fibre phase mask. The coatings used were both single and multi-layered thin films deposited on the flat side of a lapped D-shaped fibre. The various devices yielded different index sensitivities, with the single Ge coated device possessing the highest sensitivity in the aqueous regime while evidence suggests that the Ge-SiO<sub>2</sub>-Ag coated device should be the most sensitive at low indices, both devices outperforming LPGs in these regimes. Some of the SPR devices have high coupling efficiency in excess of 40dB in air and the Ge-SiO<sub>2</sub>-Ag coated device possessed an estimated index spectral sensitivity of  $\Delta\lambda/\Delta n = 90\text{nm}$  and a coupling strength sensitivity of  $\Delta I/\Delta n = 165\text{dB}$  in the index range from 1 to 1.15. A device with a single layer of Ge demonstrated an index spectral sensitivity of  $\Delta\lambda/\Delta n = 12500\text{ nm}$  over the index range of 1.36 to 1.39 with higher sensitivities obtained at higher indices.

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